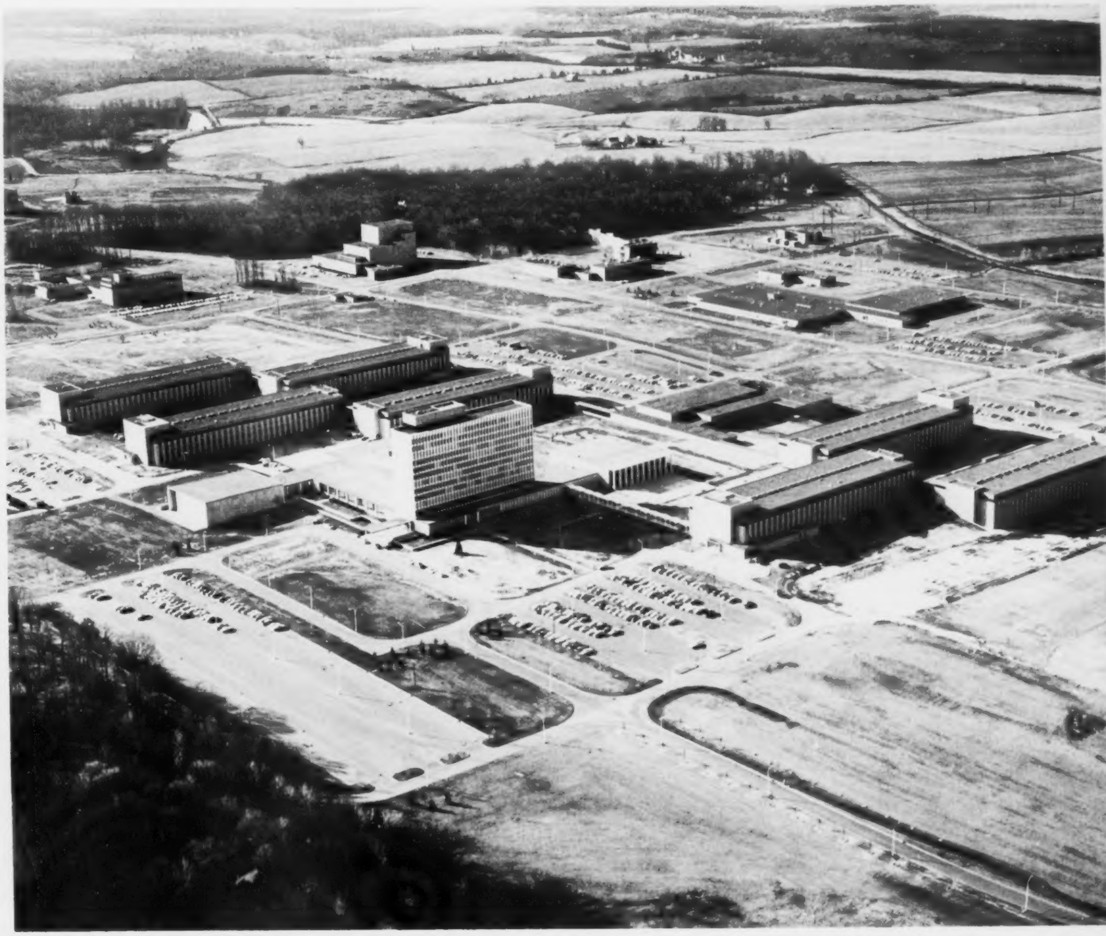
NATIONAL BUREAU OF STANDARDS February 1966

Technical News Bulletin



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News Bulletin

FEBRUARY 1966/VOL. 50, NO. 2/ISSUED MONTHLY

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COVER

New NBS Gaithersburg, Maryland, facilities now nearing completion and being occupied.
(*See Aug. 1960 issue*)

U.S. DEPARTMENT OF COMMERCE
John T. Connor, Secretary

NATIONAL BUREAU OF STANDARDS
A. V. Astin, Director

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- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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WINDS ARE NOISY...

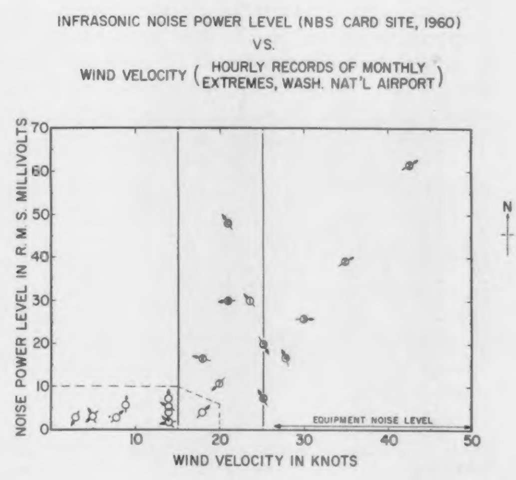
in the inaudible range

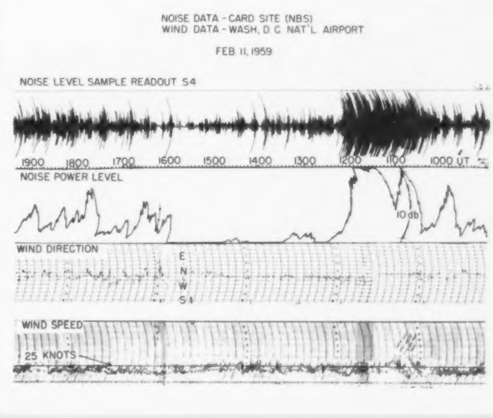
■ A direct relation between high-velocity winds and infrasonic noise in the atmosphere has been found at the NBS Institute for Basic Standards. In this work, Howard S. Bowman of the NBS acoustics laboratory compared local wind velocities measured by the U.S. Weather Bureau with infrasonic noise measurements made at NBS over the same time intervals. The comparisons showed that large changes in wind direction or peak wind gusts accompanied high infrasonic noise levels, thus indicating noisy winds even in the inaudible range.

These results are of particular interest to investigators studying the origin and direction of travel of low-frequency infrasonic signals as they are propagated through the earth's atmosphere. Such signals, generated by natural phenomena like volcanoes and earthquakes, are detected on sensitive instruments located vast distances away from the origin of the phenomena. Background noise, however, apparently caused by local disturbances, sometimes distorts or interferes with the measurements thus obtained.

The present study shows that this background noise is due to high-velocity winds in the vicinity of the measurement equipment. Hence, areas having a low probability of high winds should be designated for future infrasonic experimental sites. The study also demonstrates the need for improved wind filters on present infrasonic instruments to eliminate wind noise when high winds do occur.

Left: *Infrasonic noise power levels as a function of extremes of wind velocities for several months. The numbers inside the circles correspond to the months, and the arrows show wind direction. Data inside the dashed lines are associated with minimum noise power levels and wind velocities. The vertical lines encompass a region of poorly correlated data. Above 25 knots, however, the data show a linear relationship.* Right: *Infrasonic noise data obtained locally by NBS over a period of nine hours are compared with U.S. Weather Bureau wind data derived for the same period. The comparison shows that noise power levels, obtained by a technique devised by Howard S. Bowman, and wind velocities rise simultaneously.*





FROST IS FINGERPRINTED

■ Graphic "fingerprints" of water frost and of carbon dioxide "frost" have been obtained in recent measurements at the NBS Institute for Basic Standards. The work was undertaken by H. J. Keegan and V. R. Weidner of the NBS staff to aid studies of the Venus cloud cover, which may be composed of crystals of one or of both of these substances.[1] The Advanced Research Projects Agency of the Department of Defense and the Goddard Space Flight Center of the National Aeronautics and Space Administration sponsored the research.

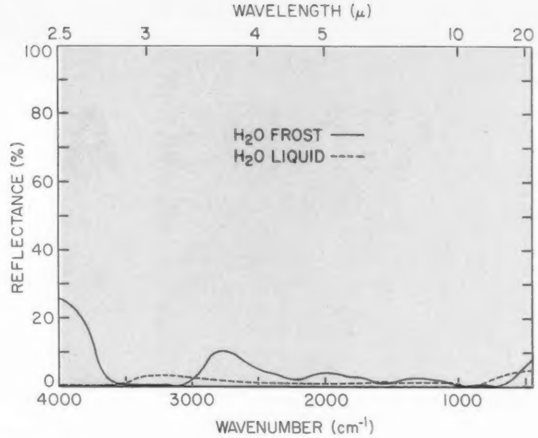
The fingerprints—clear patterns of the light-reflecting and absorbing properties of the two types of frost at wavelengths in the infrared region of the spectrum—will be checked against infrared spectra to be obtained from Venus in future space probes. They may also aid in climatic studies of the planet. For example, it is not now known why the clouds surrounding Venus apparently remain at the same temperature, both on the bright side of the cloud cover reflecting the sun's rays, and on the dark side away from these rays.

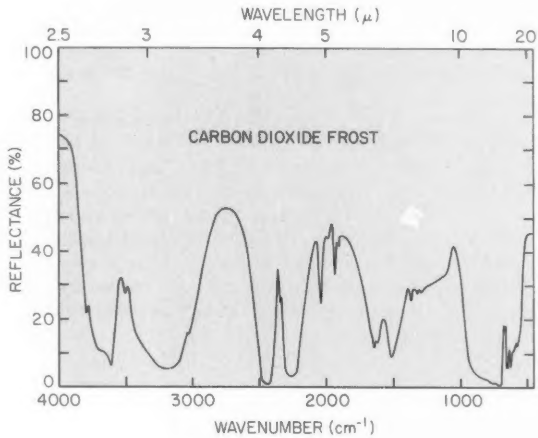
To achieve accurate measurements, the investigators used a special reflectance attachment on a high-resolution spectrophotometer.[2] The attachment excluded the radiant energy emitted by the specimens. The inclusion of such emission, which only affects spectrophotometric measurements in the infrared, would have distorted experimental results.

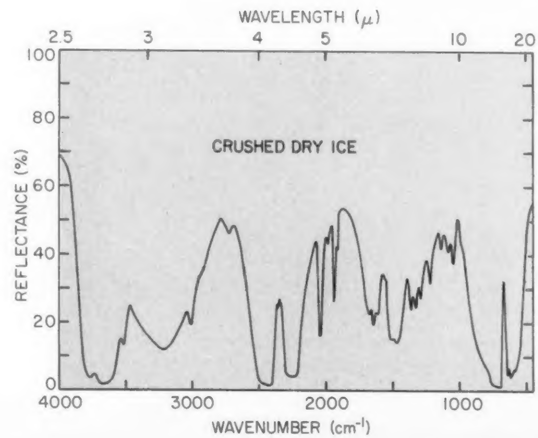
Four types of specimens were investigated: (1) Frost collected from atmospheric water vapor on a supercooled base; (2) water at room temperature—for comparison purposes; (3) carbon dioxide frost formed on a supercooled base in a closed container of evaporating solid carbon dioxide; and (4) crushed commercial "dry ice," i.e., solid carbon dioxide.

The method of specimen preparation did not rule out contamination of the water frost with the carbon dioxide frost, and vice versa; however, no common features were found in the resulting spectra of the various types of specimens. It therefore appears that the

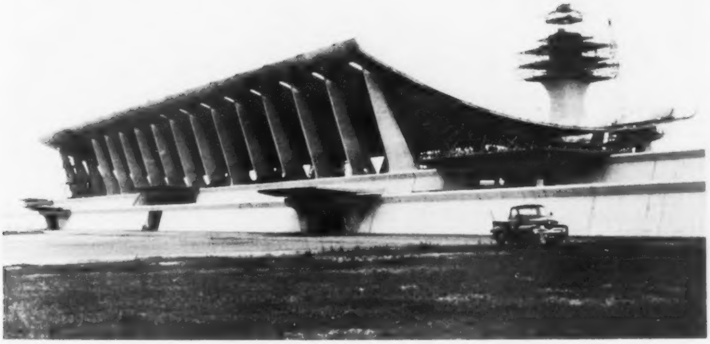
(continued on p. 22)







Top: *Infrared spectral reflectance (2.5 to 22.2 microns) of water frost (solid curve) at the boiling point of nitrogen (—196 °C). Also shown (dotted line) is the reflectance of distilled water at room temperature.* Center: *Reflectance of carbon dioxide frost at a temperature of —196 °C.* Bottom: *Reflectance of crushed "dry ice" obtained at a temperature of —78 °C (the sublimation temperature of carbon dioxide).*



assessing durability of PLASTIC ROOFS

■ In recent years a large number of flexible roof coverings have been developed from polymers and synthetic rubbers. These coverings are lightweight, highly elastic, highly reflective, and easily cleaned of radioactive fallout. They are used mainly where more conventional roofing materials may not be able to perform, as on roofs of unusual contour. Considerable interest is being shown in the properties of these new materials, especially their durability. As roofs are directly exposed to solar radiation, their resistance to photo-oxidation, a measure of their durability, is of prime concern.

Recently, a study was initiated at the NBS Institute for Applied Technology to determine the photo-oxidation resistance of some plastics and to investigate the usefulness of a recently developed colorimetric method of measuring photochemical degradation of polyesters. The results of this study by K. G. Martin* indicate that the colorimetric method studied should be a valuable means of rapidly assessing the relative stability of polymeric coatings exposed to sunlight.

The colorimetric method is based on the reaction of *N*, *N*-dimethyl-*p*-phenylenediame (DMPDA) with the photo-oxidation products that form on the surface of a wide range of plastics. By determining the amount of DMPDA that reacts with these products, the degree of photo-oxidation of the plastic can be assessed. This method is more sensitive than methods that involve the complete specimen, as both photo-oxidation and the DMPDA reaction are surface reactions only.

Flexible plastic roofing was used on the terminal building at Dulles International Airport (Chantilly, Va.) as conventional materials would not withstand the expected large roof movements (about 8 in.)

Seven commercial coatings—five liquid-applied coatings and two prefabricated sheets—were examined. The sheets were poly(vinyl-chloride) (PVC) and poly(vinyl-fluoride) (PVF); the liquids were acrylic emulsion (Ac), butyl-rubber emulsion (BR), chlorosulfonated polyethylene solution (CSP), poly(vinyl-chloride-acetate) solution (PVCA), and silicone-rubber solution (SR). Of these, BR, PVC, and PVCA were not specifically formulated for roofing use.

The liquid-applied materials were cured on glass strips into specimens 0.005- to 0.007-in. thick. Sheets of PVC and PVF, respectively 0.100-in. and 0.002-in. thick, were mounted on glass strips, also. Samples of each plastic were exposed to solar and carbon-arc radiation.

In the colorimetric method, a known amount of DMPDA in a benezene-methanol solution is placed in a container with the plastic specimen. The container is shaken for two hours, after which the specimen is removed.

During shaking, the DMPDA reacts with the plastic, changing the color of both the plastic and the solution. The irradiated specimens produce greater color changes than the unexposed specimens, with the colors ranging from yellow to brown to greenish brown.

Three techniques of judging the extent of the DMPDA reaction with a photo-oxidized plastic were found to be useful: (1) Determination of the amount of DMPDA reacted, (2) measurement of the DMPDA reagent solution color, and (3) measurement of specimen surface darkening.

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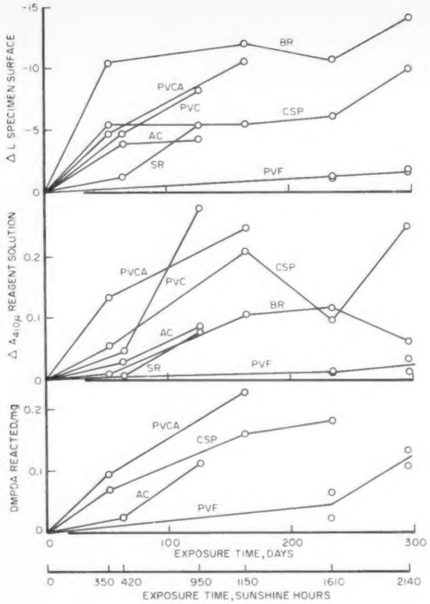


The progressive photo-oxidation of plastic roof coverings exposed to sunlight was determined three ways: (a) [top] darkening ($-\Delta L$) of the specimen surface, (b) [center] change in absorbance (ΔA) of the reagent solution at 410 mμ and (c) [bottom] the amount of DMPDA that reacted with the exposed surface. The specimens were exposed to natural weathering conditions atop a building at the Bureau in Washington, D.C.

In the first technique, samples of the reagent solution are diluted in methanol and a buffer, and treated with benzoyl peroxide. The peroxide oxidizes any unreacted DMPDA, causing the diluted solution to turn pink in cloor. The intensity of this color, determined by the absorbance at 520 nm, indicates the amount of DMPDA that was left in solution. The difference between the known amount of DMPDA originally in the solution and that left in solution is the amount that reacted with the plastic.

In the second technique, the change in the absorbance at 410 nm of undiluted samples of the DMPDA reagent solution is measured. This indicates the amount of photo-oxidation products that dissolved in solution and reacted with the DMPDA.

In the third technique, color measurements with a tristimulus differential colorimeter are made of the specimens in three conditions of exposure and DMPDA treatment: (1) unexposed and untreated, (2) unexposed and treated, and (3) exposed and treated. The first condition gives a base value for comparison with the others. The difference between conditions (1) and (2), if any, is subtracted from the difference between (1) and (3) to show the effect of exposure alone. Average values of darkening were calculated and show a direct relationship to exposure time.

The three techniques showed good agreement in ranking the plastics as to degree of photo-oxidation. Assessment of the DMPDA reaction generally agreed with the amount of darkening caused by exposure. The seven roofing materials examined were generally rated in order of durability as follows, beginning with the least stable: PVCA, PVC, CSP, BR, Ac, SR, and PVF.

Frost is fingerprinted—Con.

samples were essentially uncontaminated. Moreover, the spectra of both the carbon dioxide frost and of the crushed dry ice were strikingly similar at all significant wavelengths.

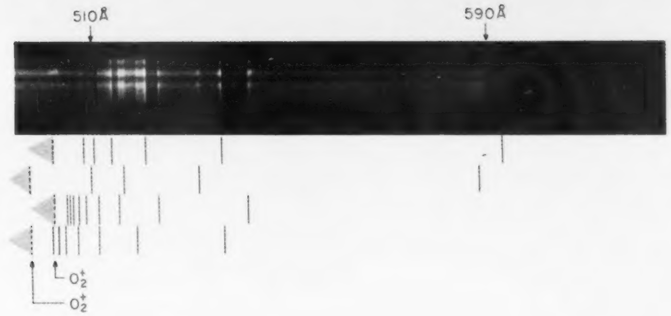
The study was carried out for Prof. John D. Strong of the Johns Hopkins University's astrophysics laboratory. Prof. Strong recently reported on measurements of the spectral reflectance of Venus obtained across part of the infrared from balloon-telescope flights in 1964.[3] He applied laboratory corrections for absorption by residual water and carbon dioxide vapor in the upper atmosphere of Venus to these measurements. From the results, he concludes that the planet's clouds are composed of ice crystals.

Water frost data obtained in the present study confirm Prof. Strong's laboratory findings, although not in complete detail. The NBS measurements, however, on both water frost and solid carbon dioxide were made over a much larger infrared region than were Prof. Strong's measurements. They therefore supply a basis for more extended checks of the composition of the Venus cloud cover in future probes.

[1] *For further details, see* Infrared spectral reflectance of frost, *by H. J. Keegan and V. R. Weidner, J. Opt. Soc. Am.* **56** *(1966).*

[2] New method for measuring diffuse reflectance in the infrared, *by J. U. White, J. Opt. Soc. Am.* **54,** *1332–1337 (1964).*

[3] Composition of venus clouds and implications for atmospheric models, *by Bottema, Plummer, Strong, and Zander, J. Geophys. Res.,* **70,** *4401–4402 (1965).*



new energy states in
MOLECULAR OXYGEN

■ New energy states in neutral oxygen, higher in energy than any previously reported for this molecule, have recently been observed [1] by Keith Codling and R. P. Madden of the NBS Institute for Basic Standards in work partly supported by the National Aeronautics and Space Administration. These new energy states form Rydberg series that lie in an energy range around 25 eV, a range of particular importance in plasma physics, atmospheric physics, and astrophysics. Their discovery provides information of value in the understanding of molecular structure.

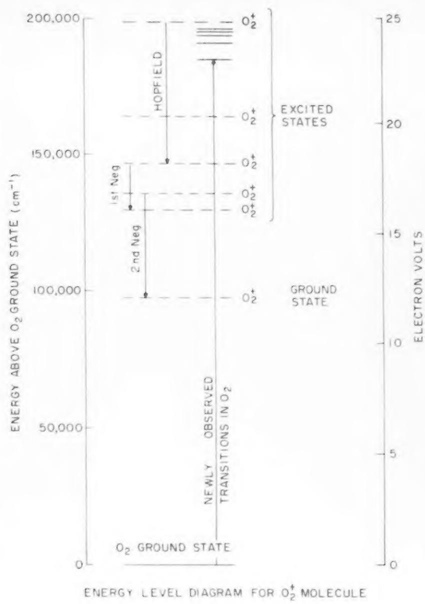
The new states undergo autoionization—a process which may occur when an atom is excited to a state above its first ionization threshold. This is accomplished by excitation of an electron occupying an inner shell, as in the present case, or by double-electron excitation, i.e., the simultaneous excitation of two of the atom's outer shell electrons. These excited states in the continuum (region above the ionization threshold), however, are very unstable (with typical lifetimes of 10^{-13} sec); this fact is reflected in the relatively broad structures observed.

The absorption spectrum of neutral oxygen in the 500–625 Å region, showing four Rydberg series. As the photograph is a positive print, black denotes absorption. Note the white features resembling broad emission lines. These actually are discrete lacks of absorption. Lines below the print show the position of series components.

The interaction of these high-lying states with adjacent continua may produce "resonances" with very unusual shapes. Depending upon the interaction, one may even observe absorption features which appear as if they were emission lines. These are referred to as "window-type" resonances, since they in fact involve discrete lacks of absorption at certain points in a continuous spectrum. The existence of these resonances of such unusual profile has been something of a mystery in the past. However, scientists are now able, in a qualitative way, to understand them.

The astrophysical importance of the autoionization process has only quite recently been appreciated. For instance, consideration of this mechanism may have a profound effect on the interpretation of spectra obtained from the sun's lower chromosphere. Also, laboratory observation of autoionized states in the extreme ultraviolet region will be of use in analyzing spectra obtained in the future from astronomical sources such as the outer (cooler) portions of nearby stars or from comet tails.

In the present work,[1] a 3-m grazing incidence spectrograph, with a 600-line/mm grating and a spectral slit width of about 0.06 Å, was used. The background source was the "light" radiated by the accelerating electrons in the NBS 180-MeV synchrotron. This radiation is continuous in wavelength, and for the NBS



Left: *Energy level diagram of the O^+_2 molecule, showing the new transitions.* Right: *Apparatus used to observe new energy states in neutral oxygen, above 20 eV. R. P. Madden (left) adjusts the automatic pressure controller on advice from Keith Codling, who has just read the pressure gage that connects directly with the interaction chamber. After the pressure has reached equilibrium, the synchrotron is operated remotely. At upper right under the dark cover is the 3-m vacuum spectrograph.*

machine is usable down to below 100 Å.[2] With this unique source, previously unobserved states of neutral oxygen have been detected. These neutral states form Rydberg series which converge, in the limit, to a state in O_2^+, where the excited electron is completely removed. The convergence limit allows the energy of this state of O_2^+, to be determined quite accurately. The existence of this state at 24.56 eV, first suggested by Leblanc,[3] has been in some doubt, but the present work definitely confirms that there is indeed a state of O_2^+ at this energy.

This work is part of a larger coordinated program of research at NBS[4] which has yielded important new information on the energy states of atoms and molecules in the so-called "intermediate" energy range—that is, in the energy range from 10–1000 eV. The NBS synchrotron, which is at present one of the most important sources of pure continuum in the energy range above 20 eV, has been used to observe many new "resonances" in photoionization continua of various atoms and molecules. Relevant theory has also been worked out by NBS theoreticians.[5]

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[1] *For further technical details see,* New Rydberg series in molecular oxygen near 500 A, *by K. Codling and R. P. Madden, J. Chem. Phys.* **42,** *3935 (1965).*

[2] Characteristics of the "synchrotron light" from the NBS 180 MeV machine, *K. Codling and R. P. Madden, J. Appl. Phys.* **36,** *380 (1965).*

[3] Electronic states of Hopfield's oxygen emission bands, *by F. J. Leblanc, J. Chem. Phys.* **38,** *487 (1963).*

[4] NBS develops new tool for exploring atomic structure, *NBS Tech. News Bull.* **48,** *4 (1964).*

[5] Effects of configuration interaction on intensities and phase shifts, *by U. Fano, Phys. Rev.* **124,** *1866 (1961).*

Standard Materials

Isotopic reference standards

Four isotopic reference standards are now available from the NBS Office of Standard Reference Materials. They are sodium chloride No. 975 for chlorine, copper metal No. 976 for copper, sodium bromide No. 977 for bromine, and silver nitrate No. 978 for silver.[1] These standards are natural-ratio materials which may serve as reference ratios for investigators looking for small variations in the isotopic composition of the elements. They will also be useful in the measurement of mass-discrimination effects encountered in the operation of mass spectrometers.

The isotopic compositions of these standards were determined [2] by triple filament mass spectrometry by comparing them to mixtures prepared from high purity isotopes. Overall limits of error are based on 95 percent confidence limits for the mean and on allowances for the effects of known sources of possible systematic error. Each standard is certified for isotopic composition and costs $20.00 for a 0.25 g unit.

TABLE 1. *NBS Isotopic Standards*

Standard/Element			
No. 975, Chlorine.	Absolute abundance ratio, Cl 35/Cl 37	3.1272	$^{+0.0079}_{-0.0082}$
	Chlorine 35, atom percent	75.770	$^{+0.044}_{-0.046}$
	Chlorine 37, atom percent	24.229	$^{+0.046}_{-0.044}$
No. 976, Copper.	Absolute abundance ratio, Cu 63/Cu 65.	2.2440	±0.0021
	Copper 63, atom percent	69.174	±0.020
	Copper 65, atom percent	30.826	±0.020
No. 977, Bromine.	Absolute abundance ratio, Br 79/Br 81	1.02784	±0.00105
	Bromine 79, atom percent	50.686	±0.025
	Bromine 81, atom percent	49.314	±0.025
No. 978, Silver	Absolute abundance ratio, Ag 107/Ag 109.	1.07597	±0.00135
	Silver 107, atom percent	51.830	±0.052
	Silver 109, atom percent	48.170	±0.052

List of available radioactive standards amended

Three point-source gamma-ray standards have been prepared to replace out-of-stock standards. NBS Standard No. 4992–C, zinc 65, costing $50 per sample, replaces No. 4992–B. NBS Standard No. 4998–C, yttrium 88, costing $53 per sample, replaces No. 4998–B. NBS Standard No. 4999–C, cerium 139, costing $58 per sample, replaces No. 4999–B.

In addition, NBS Standard No. 4964–B, a radium gamma-ray solution, replaces No. 4964. This standard has a radium content of 100 μg (1965) in a 5 g solution and costs $43 per sample. The price of each of the other radium gamma-ray solution standards, Nos. 4955, 4956, 4957, 4958, 4959, 4960, 4961, 4962, and 4963, has been increased to $34 per sample.

Three radioactivity standards are temporarily out of stock: (1) NBS No. 4932, mercury 203, (2) NBS No. 4944, iodine 125, and (3) NBS No. 4946, cerium 141. Also NBS Standard No. 4941, cobalt 57, has been discontinued, as cobalt 57 is now commercially available.

Thermal Emittance (Emissivity) Standards

Thermal emittance (emissivity) standards are required by the aerospace industry to obtain valid emittance data on structural materials used in aircraft and missiles. Disagreement on the reliability of such data hampers vehicle designers in estimating important parameters such as surface temperatures.

To meet the needs of industry, the NBS Office of Standard Reference Materials has recently made available 25 thermal emittance standards.[1] These standards have been prepared for use at three levels of emittance from three alloys: (1) polished platinum-13 percent rhodium, 0.035 in. thick, for low emittance (below 0.3), (2) sandblasted and oxidized Kanthal (an iron, chromium, aluminum alloy), 0.040 in. thick, for intermediate emittance (0.5 to 0.75), and (3) sandblasted and oxidized Inconel (a nickel, chromium, iron alloy), 0.055 in. thick, for high emittance (0.75 and above).

Similar standards were previously available as working standards from the Bureau's Building Research Division.[2] The Inconel and Kanthal standards now issued are certified for normal spectral emittance at 800, 1100, and 1300 °K, and the platinum-13 percent rhodium standards are certified at 800, 1100, 1400, and 1600 °K. Each alloy is available in the following shapes and sizes: disks (diameter in inches) ½, ⅞, 1, 1⅛, and 1¼; squares 2 × 2 in.; and strips 1 × 10 in. and ¾ × 10 in. Kanthal is also available in a strip ¼ × 8 in. The Kanthal and Inconel standards, numbered according to size from 1420 to 1428 and from 1440 to 1447, respectively, costs $175 each. The platinum standards are numbered from 1402 to 1409 and cost, respectively, $175, 185, 200, 235, 250, 385, 750, and 600.

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[1] *These standards may be ordered from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C., 20234. For a list of NBS standard materials, see* Standard Reference Materials: catalog and price list of standard materials issued by the National Bureau of Standards, *NBS Misc. Publ. 260, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, for 45 cents.*

[2] Atomic weights redetermined by mass spectrometry, *NBS Tech. News Bull.* **49**, *No. 5, p. 74 (May 1965).*

For additional information, see NBS develops methods for determining thermal radiation properties of materials, *NBS Tech. News Bull.* **47**, *No. 10, p. 185–187 (October 1963).*

799-738 O—66——2

■ The Research Associate Plan of the Bureau offers a valuable means for Government—industry cooperation. Under this plan, industrial groups sponsor research at the Bureau which is of special interest to them and yet has public significance. Experiments are carried out by Research Associates who are paid by their sponsors but who work in Bureau laboratories with the Bureau staff. The knowledge and skills thus derived by Research Associates are expected to advance the nation's economy and to increase utilization of NBS research results.

New emphasis has recently been placed on the Research Associate Plan to make the unique facilities of the Bureau more available to industry and commerce. In this way, wider use may be made of NBS instrumentation and of the measurement techniques that have been acquired by the NBS staff. The Research Associates contribute their industrial experience and their knowledge of urgent industrial needs to the success of the plan.

Robert L. Stern, head of the NBS Office of Industrial Services, is in charge of the expanding program. He arranges meetings between potential sponsors and NBS scientists to determine where a joint effort can solve a technological problem of special importance. Once the problem has been clearly defined, an agreement is entered into outlining the scope of the work and the way in which it is to be carried out. The results achieved through the combined efforts are made available through publication.

The work of Research Associates is not directed to proprietary solutions. Instead, it is aimed at removing obstacles to the use of measurement for achieving high precision. Such obstacles may arise from a lack of sound data or instrumentation, or from a lack of criteria or of appropriate techniques.

Space Fuels

As a result of the expanded plan, several specialists have joined the NBS staff. Most recent is Dale R. Nielsen, an Aerojet General Corp. engineer, who is now working in the NBS cyrogenics laboratory in Boulder, Colo. Mr. Nielsen is studying the behavior of low-temperature fuels in flow such as liquid hydrogen and nitrogen that are used in spacecraft. The findings of this study will aid in the design of fluid handling by pumps, which are now compromised by cavitation problems.[1]

Laser Research

Among areas in which the Bureau has pioneered in recent years is in the use of lasers for length measurements.[2] Instrument companies are interested in this development since it has potential applications in tool engineering. To aid in solving problems such as time instability in laser emission, Dr. Richard Zipin of the Sheffield Corp., has recently joined the NBS metrology laboratory as a Research Associate. The results of his work may point the way to the industrial use of laser-equipped instruments for making machine tools.

NBS broadens
COLLABOR[A]
INDU[S]

Research on Construction Materials

Two industrial chemists have been appointed under the expanded plan to study the processes involved in the weathering of plastics. They are Dr. William F. Brucksch of the U.S. Rubber Co., and Dr. Joseph F. Clark of the Grace Chemical Co. Their work at NBS is being sponsored by members of the Manufacturing Chemists' Association.

The purpose of this work is to develop improved methods for predicting the weathering of plastic compositions under outdoor exposures. The research is complementing the efforts of the NBS building research laboratory where colorimetric techniques have been devised for measuring the accelerated degradation of plastics.[3]

Microfilm Research

Donald R. Lehmbeck, a new Research Associate in the NBS photographic research laboratory, is sponsored

Left: *Richard Zipin adjusts a laser used in making interferometric measurements of length.* Center: *Dale R. Nielsen works in the NBS cryogenic laboratory at Boulder, Colo. Here he is checking instrumentation used in cavitation studies of low-temperature fuels.* Right: *Joseph F. Clark (top) on a laboratory roof adjusts a pyroheliometer that detects solar radiation as William F. Brucksch (bottom) on the floor below examines a recording of the radiation.*

ORATION WITH DUSTRY

by the National Microfilm Association. Mr. Lehmbeck is investigating the structure of blemishes that sometimes form on microfilm and the mechanism of their formation.[4] Techniques are being sought in this study to prevent blemish formation.

Fire Protection

Dr. John Rockett and Lawrence Orloff are two Research Associates recently appointed in the NBS fire research laboratory. They are the first appointments to a major program of basic fire research sponsored by the Factory Mutual System. They have begun a study of thermally induced convection in a room. Other areas of study, concentrated on the early stages of the development of a fire, will be added as the program gains momentum. The results of this basic work will aid in the design of fire detection and suppression systems for large structures such as factories and office buildings.

Left: *Donald R. Lehmbeck makes preliminary adjustments to a microtone recently installed in the NBS photographic research laboratory. The instrument is being used to develop techniques for sectioning microfilm specimens for subsequent electron microscope studies.* Right: *Lawrence Arloff (left) and John Rockett (right) discuss equipment recently installed in the NBS fire research laboratory. This equipment is used to obtain a better understanding of the behavior of flame inhibitors.*

Additional Opportunities

Many opportunities for Research Associate work are available at the Bureau. Some of the interesting areas in which such work has an immediate potential are: Analytical chemistry, including activation analysis and Mössbauer effects; crystal growth techniques for metals, ceramics, and polymers; precision measurements of humidity, pressure, vacuum, nuclear, and particle radiation, and microwave pulse-voltage and power; determination of material properties in high-frequency electrical and magnetic fields; spectroscopy over the range from microwave through infrared up to X-ray frequencies; criteria development for the performance of engineering devices and systems; systems design for technical information dissemination; and standards development for computer languages and data processing systems.

Industrial or professional groups that are interested in sponsoring Research Associate work at the Bureau should get in touch with Mr. Robert L. Stern, National Bureau of Standards, Washington, D.C., 20234.

[1] NBS studies problems of handling space fuels, *NBS Tech. News Bull.* **49,** *191* (*1965*).

[2] Laser provides accurate length measurements, *ibid.* **49,** *10* (*1965*).

[3] *For a brief treatment of this work, see* Oxidation products of degraded plastics identified, *ibid.* **48,** *83* (*1964*), *and* Colorimetric determination of plastic degradation, *ibid.* **47,** *64* (*1963*).

[4] Current research on preservation of archival records on silver-gelatin type microfilm in roll form, *by C. S. McCamy and C. I. Pope, J. Research NBS* **69A** (*Phys. and Chem.*) *385* (*1965*).

OXYGEN CONTAMINATION

on platinum surfaces

■ The field-electron microscope is one of the most useful tools available to scientists for the study of clean surfaces. With this instrument, contaminants (such as oxide layers and other impurities which adhere to all metals exposed to air) can be removed *in vacuo* by purely electrical means and the attainment of a perfectly clean surface can be verified visually.

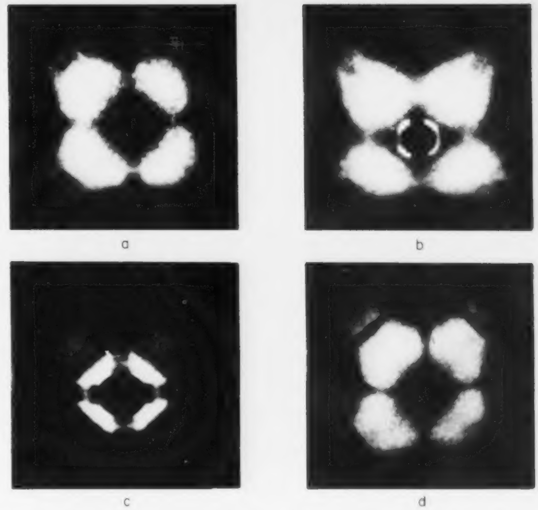
In the case of platinum, however, the presence of a tightly bound contaminant in the neighborhood of the {001} planes has been noticed by various experimenters and has hindered surface investigations. A recent study by Allan J. Melmed of the NBS Institute for Materials Research has shown this contaminant to be oxygen (or oxide).[1] The results of this work, which was sponsored by the Advanced Research Projects Agency, will make possible more comprehensive field-electron microscope studies of clean, single-crystal platinum surfaces.

The platinum field-emitter tips used in this experiment were made by spotwelding 0.002-in. diam. platinum wires to platinum support loops. The wires were then electrolytically etched and polished in an aqueous solution of potassium cyanide or potassium chloride at room temperature. Of the tips that were made, some were spotwelded in a nitrogen atmosphere and some were spotwelded in air.

A vacuum-processed field-electron emission microscope baked at about 420 °C was used in the investigation. The final ambient pressure of the system was less than 1×10^{-10} torr.[2] The emitter tip was heated for several seconds at progressively higher temperatures and cooled to room temperature between heatings to make field-electron emission observations.

At first the electron emission patterns were irregular, but after heating to 1100–1200 °C a regular pattern was obtained which appeared to represent the emission from a clean surface. However, heating to a higher temperature caused the appearance of bright rings or ring sections around the {001} planes. This contamination was called condition "A." Further heating (above 1200 °C) produced a more extensive and complex bright structure around the {001} planes. This further degree of contamination was called condition "B."

For the emitters spotwelded in nitrogen, the "B" condition could be changed to the "A" condition and then to a clean surface by heating to 1500–1600 °C for about 1 minute. Also, a return to the clean surface condition from the "A" condition could be achieved by heating to about 1100 °C for a few minutes. For the emitters spotwelded in air, however, it was difficult or impossible to clean the tip in the time available (1-3 hours) before excessive blunting occurred due to the heating. It was also found that if the emitter tip was



Field-electron micrograms of platinum. Study has shown (001) plane contamination to be oxygen or oxide. All patterns have (001) plane in the center. (A) Clean surface appearance after initial heating to 1100–1200 °C in vacuum. (B) Contamination condition "A". (C) Contamination condition "B". (D) Clean surface after heating to 1500–1600 °C in vacuum.

heated to about 1000 °C in air prior to installation in the field-electron microscope, the contamination could never be thermally removed.

A clean surface emitter tip was then heated to 1000 °C in an atmosphere of ultrapure oxygen, diffused into the vacuum system through hot silver. In every case, condition "A" or "B" was produced depending on the amount of oxygen exposure. It was found that for small oxygen exposures the method of heating for one minute at 1500–1600 °C cleaned the surface but that for large exposures the tip could not be cleaned.

It was concluded that the contamination on the {001} planes is due to tightly bound oxygen (or oxide) contained in solution in the bulk platinum. The difficulty in removing the oxygen increases with the amount dissolved, and therefore in some cases, depending on the history of the emitter, it is impossible to achieve a clean tip surface. If too much oxygen (or oxide) has diffused into the platinum, the heat required to clean it is so great that the tip will blunt beyond use.

Contaminated tips were heated in hydrogen and carbon monoxide atmospheres in attempts to remove the contamination by chemical reduction. In both cases the surface contamination was reduced drastically, often completely, but it returned upon subsequent heating in vacuum.

Experimentation indicated that oxygen contamination might be removed completely by repeated reduction, but a simpler procedure would be to avoid heating the platinum in an oxidizing atmosphere, thus avoiding initial contamination.

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[1] *For further technical details, see,* An interaction of oxygen with platinum, *by Allan J. Melmed, J. Appl. Phys. (in press).*

[2] *1 torr=1/760 standard atmosphere=133.322 newton/meter*^{2}.

Oxygen contamination—Con.

Allan Melmed prepares to photograph a field-electron emission pattern which appears on the powder screen of the field-emission tube (center in front of camera).

Gamma-Ray and Neutron Reflection

■ A technique for calculating the reflection of gamma rays or neutrons from a surface has been devised by C. Eisenhauer of the NBS Institute for Basic Standards.[1] The calculation uses a simplified representation for the problem of a point source and a point detector located above a semi-infinite slab of reflecting material; the reflection is shown to be primarily dependent on a single variable, with only a slight dependence on a second variable.

Reflected radiation is of concern in any radiation measurement situation. Floors, walls, adjacent equipment, and a variety of other objects act as reflectors of radiation, and laboratory measurements must be corrected for radiation scattered in this manner. Also, since radiation shields such as lead and concrete reflect as well as absorb, the accurate measurement of reflected radiation is of importance in designing equipment and in planning safety measures.

Mr. Eisenhauer found that the main features of the reflected radiation problem are determined by the geometry of the source and the detector, and that this geometry can readily be evaluated by studying specular (mirror) reflection—the most common type of reflection phenomenon. He studied the simple case of Lambert reflection[2] and concluded that once the geometric effects are determined the remaining behavior can be expressed by the cosine of the angle defined by specular reflection.

This analysis was then extended to include the reflection of cobalt 60 gamma rays from concrete; it was found that such reflection can be expressed as primarily a function of the cosine of the specular angle, with only a secondary dependence on the ratio of the radiation detector height to the source height. This conclusion was shown to be reasonable by comparing the calculation results with experiments and independent calculations for a cobalt 60 source (1.17 and 1.33 Mev gamma rays) and a polonium-beryllium source (zero to 10 Mev neutrons).

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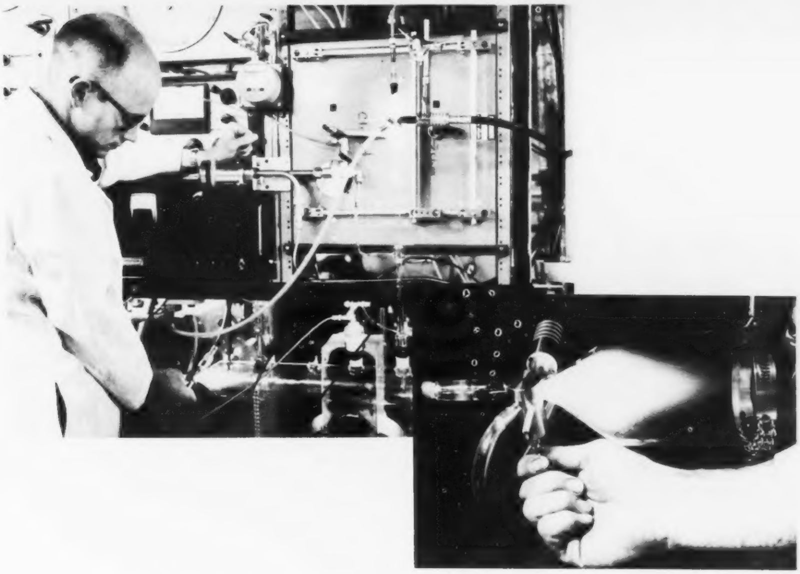
[1] *For further technical details, see* An image source technique for calculating reflection of gamma rays or neutrons, *by C. Eisenhauer, Health Phys. J.* **11**, *p. 1145 (1965).*

[2] *Lambert reflection obeys the Lambert Cosine Law, which states that the intensity from a surface element of a perfectly diffuse radiator is proportional to the cosine of the angle between the direction of emission and the normal to the surface. An element of the surface which obeys the law will appear equally bright when observed from any direction.*



NEW MICROWAVE DISCHARGE CAVITIES

■ Two microwave discharge cavities that offer many advantages in atomic and molecular research have been developed by K. M. Evenson, H. P. Broida, and F. C. Fesenfeld* of the Bureau. These cavities were constructed and evaluated at the Boulder (Colo.) Laboratories of the U.S. Department of Commerce in work supported in part by the Advanced Research Projects Agency and the Office of Naval Research.[1] The new cavities offer high efficiency and added convenience, producing high ionization with minimum heating, contamination, and electrical interference.

Microwave Discharge Cavities

Discharges in microwave cavities are useful as a means of excitation for studying gaseous electronics, producing light, and obtaining free radicals. The cavity consists essentially of an enclosure which contains the electric field in which a sample gas flows in a glass tube. The electromagnetic power applied to the cavity is increased until the sample gas becomes excited, a condition that is indicated by a glow emanating from the flowing gas. Many discharge cavities are powered by converted surplus military radars and some recent ones are powered by medical diathermy units which supply as much as 125 watts of power at 2450 MHz.

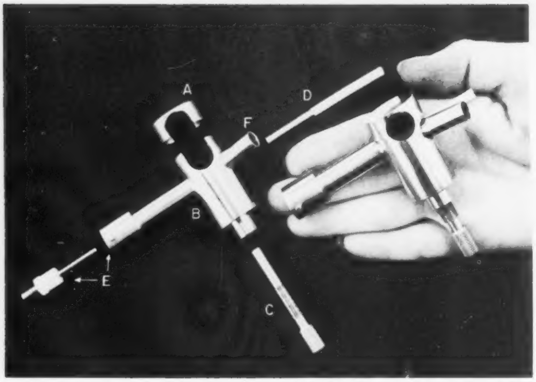
The discharge cavities available to the scientist vary greatly in their versatility and usefulness in specific programs. Some types permit adjustment of both resonance frequency and coupling to the microwave power source. Cavities vary in the pressures at which they will operate (from less than 0.001 torr to nearly atmospheric pressure) and in their efficiency (as great as 99 percent). Equally important, some of the new types of cavities can be positioned around or removed from the discharge tube without parting the tube, which destroys the vacuum and introduces contaminants.

New NBS Cavities

Both cavities developed by NBS are fed microwave energy by coaxial cable and are capable of being "matched" to the microwave source—that is, adjusted for maximum coupling of energy. One "cavity" is actually a coaxial conductor matched by positioning

Left: *Kenneth Evenson adjusts a microwave discharge cavity to produce a glow in the helium flowing into the large tube at the bottom. A second cavity is in operation at the upper right; both are stimulated by a radio frequency of 2450 MHz.* Right: *The cavity is being tuned for maximum brightness at 2450-MHz excitation. The afterglow and ionization produced by excitation within the cavity are useful in laboratories.*

**Mr. Fesenfeld is a member of the staff of the Institute for Telecommunications Sciences and Aeronomy (formerly the NBS Central Radio Propagation Laboratory) of the Department of Commerce's Environmental Science Services Administration.*



New microwave discharge cavities can be opened to accept tubing carrying the discharge gas, without loss of vacuum or introduction of contaminants. The array of its parts shows the cap (A) *and body* (B) *through which the tubing passes, tuning screw* (C), *coupling adjustment slider* (D), *coaxial connection for input radio-frequency energy* (E), *and a tube for the entrance of cooling air* (F).

a matching stub located on the coaxial connector. This "cavity" is not a resonant structure and hence is not restricted in use to a limited band of frequencies. It has worked well over a 1000-MHz band. Efficiency is particularly good at low sample pressures; reflection is less than 1 percent in helium at 0.1 torr. The discharge tube is placed in open-ended slots on opposite sides of the cavity, and the cavity is closed by a tight-fitting cap which covers the slots. When in use the discharge tube is cooled by pumping air into the cavity.

The other cavity is resonated at about 2450 MHz by means of a tuning stub and matched to the source by a coupling slider. These are interacting adjustments which, if reset alternately, result in high efficiency over a wide frequency range. Care in making these adjustments can result in reflection of as little as 1 percent of the input power for a wide variety of sample gas pressures. This cavity, like the other, can be opened by a cap for insertion and removal of the glass tube carrying the sample gas.

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[1] Microwave discharge cavities operating at 2450 MHz, *by K. M. Evenson, Rev. of Sci. Inst.* **36,** *294–298 (1965).*

Garstang Heads JILA

■ Professor Roy H. Garstang, an astrophysicist on the faculty of the University of Colorado Department of Physics and Astrophysics and a fellow of the Joint Institute for Laboratory Astrophysics (JILA), has been elected chairman of the Institute effective January 1, 1966, retiring chairman Lewis M. Branscomb announced recently.

The Institute is a joint undertaking of the University and the Bureau on the CU Boulder campus, devoted to research and graduate study in atomic physics, astrophysics, and related sciences. Leadership of the JILA is shared by its permanent senior staff, the fellows, who elect a chairman for a rotating two-year term.

Branscomb also announced the election of the five-man executive committee of the fellows, which shares with the chairman responsibility for guiding the day-to-day affairs of JILA. In addition to the chairman, Garstang, and the chief of the NBS Laboratory Astrophysics Division, Prof. Adjoint Branscomb, the committee includes Dr. Stephen J. Smith, Dr. Richard N. Thomas, and Dr. John Hall.

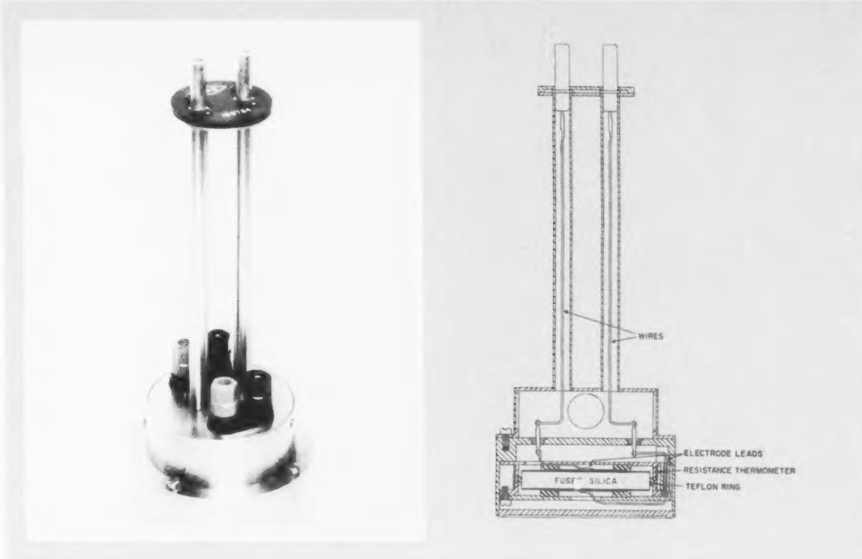
In announcing the election of Garstang to the chairmanship, Branscomb described him as a "dedicated teacher and a distinguished scientist whose research defined the meaning of the term 'laboratory astrophysics.'" Garstang came to CU last year from the Mill Hill Observatory of the University of London, where he was assistant director. He has written many research papers in atomic spectroscopy as applied to astronomical studies. According to Branscomb, Garstang's work has been of great importance in the determination of cosmic abundance of the elements, the source of much of our knowledge of the probable origin of the universe.

At CU Garstang also serves as chairman of the astrophysics committee of the Department of Physics and Astrophysics, in which astronomers in JILA and in the High Altitude Observatory of the National Center for Atmospheric Research (NCAR) are now brought together to offer a single doctorate curriculum in astrophysics.

As chairman of JILA, Garstang heads a faculty of 24, of whom 10 are distinguished visiting fellows of JILA who come on one-year appointments from around the world. In addition 11 postdoctoral research associates and 33 graduate students are working at JILA on a variety of theoretical and experimental studies in the astrophysical sciences.

In JILA's three and one-half years of existence, its staff members have published approximately 180 research papers, Branscomb said. In addition to their research, JILA faculty members carry their full share of classroom instruction, to which the NBS staff members of JILA have contributed 108 semester hours of teaching. The first doctorate awarded a graduate student in JILA was conferred last June. Two other students are expected to complete their doctorates next June.

The Institute will occupy a new building on the CU campus, which is scheduled for completion in July 1966.



PORTABLE CAPACITANCE STANDARD

■ A portable 10-picofarad capacitance standard that is both rugged and extremely stable has been developed at the Bureau by Robert D. Cutkosky and Lai H. Lee. Twelve such standards have been constructed and are presently being used in comparisons of the Bureau's calculable gage-block capacitor with other absolute standards. The portable standards simplify comparison of the accuracies of capacitance measurements at the Bureau with those of other national laboratories.[1]

The capacitance value of the portable standards is assigned on the basis of the value of the NBS absolute capacitance standard.[2] Thus a comparison between one of the portable standards and an absolute standard maintained at another laboratory serves as a check on the accuracies of the capacitance element at both laboratories. In addition, the portable capacitance standard is useful for calibrating working laboratory standards.

The design of the portable capacitance standard is similar to that of a portable capacitor built at NBS in 1961.[3] The standard, however, is smaller, lighter, and more stable than the 1961 capacitor. The increased stability was achieved by improvement of the capacitance element and its mounting.

Right: *Cross section of NBS fused-silica-dielectric capacitor. Internal shields to isolate leads are not shown.* Left: *Assembled NBS 10-picofarad capacitance standard. This rugged, portable (26 cm high by 10 cm diameter) capacitor, simplifies comparison of the accuracies of capacitance measurements at the Bureau with those at other national laboratories.*

A seven-month evaluation carried out at NBS on eleven of the twelve portable standards shortly after their completion (evaluation of these standards on a regular basis continues to the present time) showed that the order of stability of the standards, 1 part in 10^7, was considerably higher than that of the 1961 capacitors. The twelfth standard was shipped by air express to the National Research Council in Canada; a capacitance change less than 2 parts in 10^7 was observed after shipment. Additional tests are planned to establish whether the capacitors can be sent safely by mail from one laboratory to another.

The overall height of the standard is 26 cm. The capacitance element itself is 7-cm diameter and 1-cm thick. A stainless-steel vacuum-tight chamber, 10 cm in diameter and 7-cm high, houses the element in its cell. Wires from the electrodes of the element are protected 19 cm beyond the housing by stainless-steel tubes which terminate in coaxial fittings. (See illustration.)

The capacitance element is a fused-silica disk with silver electrodes fired to the two faces of the disk. The face electrodes are attached electrically to two coaxial connectors. A third silver electrode, fired onto the circumference of the disk, is grounded. The capacitance of interest is the three-terminal capacitance through the dielectric (the disk) between the face electrodes with the circumference electrode grounded.

The disk of the capacitance element is made of a high-purity optical-grade fused silica to minimize the de-

To determine the stability of the new NBS portable capacitance standards relative to each other, each capacitor in turn is measured against one "dummy" capacitor (this capacitor is not one of the portable standards) by means of a bridge. Here, Lai H. Lee is making the connection from the junction block of the bridge to one of the standards. These standards are maintained in a 25° C constant temperature oil bath.

pendence of capacitance on the frequency of the applied voltage. The thickness of the face electrodes is kept within a certain tolerance to minimize the dependence of the capacitance on the applied voltage. Finally, the assembled element is mounted in its cell in such a way as to practically eliminate the effect of mechanical shock on the capacitance.

The cell which contains the capacitance element also contains a resistance thermometer. This built-in thermometer simplifies interlaboratory comparisons since the relation between capacitance and thermometer resistance is determined at the time the standard is completed at NBS. Thus it is unnecessary for other laboratories to make precise temperature measurements in order to utilize these standards.

Details of Construction

The capacitance element was constructed by grinding the fused-silica disk to the desired dimensions and then firing three coats of a commercial silver paint at 480 °C onto the disk to form electrodes. The element was baked at 175 °C in a vacuum furnace for 24 hours to remove water from the system and then installed in its cell.

A fitted polytetrafluoroethylene (PTFE) ring around the disk supported the element in its cell. Another PTFE ring (diameter smaller than that of the disk) was placed against each disk face so that the three rings fixed the element in the cell and cushioned it against the effect of mechanical shock.

After baking, the element was cooled to 25 °C, and at this temperature, was measured and adjusted to within 50 parts per million of nominal value (10 pF). This adjustment was made by grinding a cavity in the dielectric with a small diamond wheel. The cavity was then sprayed with silver paint and refired. Generally, three successive operations were required to adjust a capacitor to within 50 ppm of nominal value.

After the capacitor element had been properly adjusted, the capacitor housing was welded shut. The completed capacitor was then evacuated through a seal-off tube and baked at 65 °C. (It was found that baking removed water from the fused silica and greatly reduced the phase angle of the capacitor.) The capacitor was filled with dry nitrogen, sealed, cycled between 0 °C and 50 °C a few times, and placed into operation.

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[1] *For further details, see* Improved ten-picofarad fused silica dielectric capacitor, *by Robert D. Cutkosky and Lai H. Lee, J. Res. NBS* **69C** *(Engineering and Instrumentation), No. 4 (July–August 1965).*

[2] Evaluation of the NBS unit of resistance based on a computable capacitor, *by Robert D. Cutkosky, J. Res. NBS* **65A** *(Phys. and Chem.), No. 3 (May–June 1961).*

[3] The construction and behavior of a transportable ten-picofarad capacitor, *by Robert D. Cutkosky and Lai H. Lee, to be published in Comité Consulatif D'Electricité, Aupres Du "Comité International Des Poids et Mesures," 10 Session 1963.*

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Technical News Bulletin, Volume **50,** No. 1, January 1966. 15 cents. Annual subscription: $1.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $5; foreign, $6. Single copy, 75 cents.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.75; foreign, $3.50. Single copy, 75 cents.

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